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**Patentanmeldung Nr.    Patent application No.    Demande de brevet n°**

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Der Präsident des Europäischen Patentamts;  
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets  
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Method and apparatus for preparing and supplying catalyst slurry to a  
polymerization reactor.

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## Method and apparatus for preparing and supplying catalyst slurry to a polymerisation reactor

### Field of the invention

5 This invention relates to catalytic reactions. In a first aspect, the invention relates to an apparatus for preparing and supplying catalyst slurry to a polymerisation reactor. In another aspect, the invention relates to a method for optimising the supply of catalyst to a polymerisation reactor.

### 10 Background

Polyethylene (PE) is synthesized via polymerising ethylene ( $\text{CH}_2=\text{CH}_2$ ) monomers. Because PE is cheap, safe, stable to most environments and easy to be processed polyethylene polymers are useful in many applications. According to the synthesis methods, PE can be generally classified into several types such as LDPE (Low Density Polyethylene), LLDPE (Linear Low Density Polyethylene), and HDPE (High Density Polyethylene). Each type of polyethylene has different properties and characteristics.

15 It is known that the polymerisation of olefins e.g. ethylene, especially by a gas phase polymerisation process, involves the polymerisation of olefin monomer with the aid of catalyst and optionally, if required depending on the used catalyst, a co-catalyst. Suitable catalysts for use in the production of polyolefins, and in particular for the preparation of polyethylene, comprise chromium-type catalysts, Ziegler-Natta catalysts and metallocene catalysts.

20 US 3,726,845 describes the supply and control of the amount of catalyst and the maintenance of the catalyst line and pump free by alternately feeding catalyst slurry and diluent to the reaction zone.

It is well known that the polymerisation reaction is quite sensitive to the quantity of catalyst utilized. It is important to control catalyst flow to a reactor since unexpected or uncontrolled catalyst injection in a reactor could lead to runaway reactions. However, one of the major problems in the injection of catalyst slurry to a reactor in prior art methods is that it is difficult to control the amount of catalyst and the flow rate of the catalyst injected.

30 GB 838,395 relates to a process and apparatus for producing a slurry of a solid catalyst in hydrocarbon diluent for use in a chemical reaction. The process comprises preparing

concentrated catalyst slurry in a hydrocarbon diluent and admixing said concentrated slurry with additional diluent and introducing said admixture to a reaction zone. According to the process, the specific inductive capacity of the slurry is continuously determined prior to the introduction of same to said reaction zone, the inductive capacity of the slurry being  
5 dependent upon the concentration of catalyst in the slurry.

US 5,098,667 describes a method for supply of a catalyst in general to a reactor comprising preparing heavy slurry in a storage vessel, and then transferring the heavy slurry to a mixing vessel e.g. by means of a metering valve such as a ball check valve, where the heavy slurry  
10 is diluted and subsequently transferred to a reactor. In the described method the flow rate of the diluted slurry is manipulated so as to provide a desired flow rate of solid particles contained in the diluted slurry. Continuous catalyst flow is maintained at a desired rate in response to a computed value of the mass flow rate of the solid catalyst particles contained in the dilute slurry. The computed mass flow rate of catalyst particles is based upon "on line"  
15 measurements of density and flow rate of the dilute catalyst slurry stream flowing to the reactor, and on predetermined densities of the solid catalyst particles and the liquid diluent constituting the slurry.

However, although the above-described methods provide an improvement on the control of  
20 catalyst flow, they have the disadvantage that the catalyst flow rate can not be reliably adjusted in function of the reaction conditions in the polymerisation reactor.

Furthermore, direct feeding of catalyst slurry to a reactor has the disadvantage that the feeding rate of the catalyst to the reactor cannot be adequately controlled. Also, in cases  
25 involving direct supply of a catalyst to a reactor, the catalysts can completely be flushed in the reactor, when a problem occurs during the preparation of the catalysts. Such uncontrolled catalyst supply may induce runaway reactions in the reactor.

Moreover, in the case catalyst in oil suspension is provided directly to a reactor, the used  
30 pumps, generally progressive cavity pumps, are not able to dose the catalyst flow and the amount of catalyst injected in the reactor. Furthermore, such systems require the switch over of the catalyst injection system, every time a new batch of catalyst needs to be connected to the reactor for supply thereto. Therefore, such injection systems do not provide an optimal and reliable control of the catalyst flow rate.

In view hereof, it can be concluded that there remains a need in the art for providing an improved method for controlling catalyst supply to a polymerisation reactor.

5 It is therefore a general object of this invention to provide an improved method for optimising catalyst introduction in a polymerisation reactor. It is a particular object of the present invention to optimise the supply of a catalyst, commercially provided in an oil suspension, to a polymerisation reactor wherein polyethylene is prepared. More in particular, the present invention also aims to provide a method enabling to effectively control the flow rate of a  
10 catalyst to a polymerisation reactor wherein polyethylene is prepared.

Furthermore, the present invention aims to provide an apparatus for preparing catalyst slurry, and for supplying said catalyst slurry to a polymerisation reactor in a controlled and efficient way.

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#### **Summary**

In a first aspect the present invention relates to an apparatus for preparing and supplying catalyst to a polymerisation reactor comprising

20 a vessel suitable for containing concentrated catalyst slurry comprising catalyst solid particles suspended in a mineral oil,

a buffer vessel for diluting said catalyst slurry at a suitable concentration for use in a polymerisation reaction, said buffer vessel being in connection with said vessel by means of one or more conduits for transferring the concentrated catalyst slurry from said vessel to the buffer vessel and being provided with one or more conduits suitable for transferring the  
25 diluted catalyst slurry from said buffer vessel to said reactor,

a pump provided on each of said conduits for transferring catalyst slurry from said vessel to said buffer vessel and

a pump provided on each of said conduits for transferring diluted catalyst slurry from said buffer vessel to said reactor.

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The present invention provides an apparatus, which allows preparing catalyst slurry having a suitable concentration for use in a polymerisation reaction, starting from catalyst, which is generally commercially supplied in a mineral oil, heptane or hexane suspension. Sometimes it is supplied in dry form.

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According to the present invention, the catalyst is not introduced directly from the catalyst transport vessel to the reactor. The apparatus further comprises an intermediate vessel, which acts as a "buffer" between the catalyst transport vessels and the reactor. The buffer vessel is operated at a pressure lower than the reactor pressure, thus eliminating the risk of uncontrolled high catalyst injection to the reactor. Furthermore, such buffer vessel enables to dampen the discontinuous catalyst feed fluctuations to the reactor. Another advantage of providing a buffer vessel is that catalyst slurry can be further diluted to a concentration suitable for use in the polymerisation reactor and that slurry having a desired, substantially constant, concentration can be prepared. Moreover, a suitable, relatively low, concentration of catalyst, preferably comprised between 0.1 and 10% by weight, will enable the use of membrane pumps for injecting the diluted catalyst slurry in the reactor. Using of diluted catalyst slurry has the advantage that it is easier to control the amount and the flow of injected catalyst.

The present invention provides an apparatus that enables to transfer concentrated catalyst slurry from the vessel to the buffer vessel before supplying the slurry to the reactor. Therefore, in another preferred embodiment, an apparatus is provided according to the invention wherein a pump is provided on the conduit for transferring concentrated catalyst slurry from the vessel to the buffer vessel which preferably comprises a progressive cavity pump. Such type of pump is particularly suitable for pumping significant amounts of solids, e.g. catalyst solid particles in mineral oil.

Moreover, the present invention provides an apparatus that enables to adjust catalyst flow to said reactor in function of the polymerisation reaction in said reactor. Therefore, in another preferred embodiment, the pump provided on the conduit for transferring the diluted catalyst slurry from said buffer vessel to said reactor comprises a membrane pump. Such pumps have the advantage that they allow the control of catalyst flow rate. Furthermore, such pumps can in particular be regulated in function of the concentration of a reactant in said reactor. Via a feed back mechanism, the membrane pumps are capable of being adjusted and of fine-tuning the catalyst flow rate to the reactor in function of the concentration of a reactant in said reactor.

In addition, the present apparatus has the advantage to be usable for different batches of catalyst. The apparatus does not need to be replaced every time a new commercial vessel comprising catalyst is to be connected to the system.

Also, the present apparatus is particularly suitable for being used for different types of catalysts, e.g. for chromium -type, metallocene as well as Ziegler -Natta catalysts.

- 5 The present invention further relates in another aspect to a method for optimising catalyst supply to a polymerisation reactor comprising the steps of
- a) transferring concentrated catalyst slurry from a vessel to a buffer vessel, said concentrated catalyst slurry comprising catalyst solid particles suspended in a mineral oil,
  - 10 b) diluting said catalyst slurry in said buffer vessel by supplying a suitable diluent in said buffer vessel, whereby diluted catalyst slurry is obtained having a suitable concentration for use in a polymerisation reaction, and
  - c) transferring said diluted catalyst slurry from said buffer vessel to said reactor at a suitable flow rate.

15 The present invention provides an improved method for injecting catalyst that is commercially supplied as solid particles, to a polymerisation reactor. Therefor, the method broadly involves the transfer of a concentrated catalyst slurry to a buffer vessel wherein catalyst is diluted and kept at a suitable concentration, before it is injected in the reactor. The method does not  
20 involve direct injection of catalyst from a (transport) vessel to a reactor.

The present invention provides in particular a method that enables to supply catalyst slurry to a reactor at a perfectly controllable flow rate of catalyst supply. The present method comprises controlling the suitable flow rate of said catalyst slurry to said reactor by  
25 determining the concentration of a reactant in said reactor. Advantageously the present method enables to fine-tune catalyst supply to a reactor in function of the polymerisation reaction in the reactor. The polymerisation production rate in the reactor can be controlled by controlling the rate of catalyst feed to the reactor. According to this aspect the reactor is fed with an adequate and optimal concentration of catalyst slurry at a suitable feed rate, and as a  
30 consequence the productivity in the polymerisation reactor and consistency of the polymerisation product are considerably improved. Fluctuations in the properties and quality of the polymerisation product resulting from the polymerisation reaction are substantially avoided. Practically, fine-tuning of catalyst supply to a reactor in function of the polymerisation reaction is enabled by providing the conduit connecting the buffer vessel to the reactor with

pumps, preferably membrane pumps, which are controllable and adjustable in function of a reactant concentration in the reactor.

5 The present invention thus provides an apparatus and method for optimising the polymerisation reaction in a reactor by optimising the process of catalyst supply to said reactor and by providing a apparatus for doing so, which is simple in design, rugged in construction and economical to manufacture. The term "optimising the polymerisation reaction" refers to the improvement of the efficiency of the polymerisation reaction and/or to the improvement of the quality of the obtained polymerisation product.

10 The method and the apparatus according to the invention are particularly useful in the polymerisation process of ethylene, and preferably in a process for preparing bimodal polyethylene.

15 The various features which characterize the invention are pointed out with particularity in the claims annexed to and forming a part of this disclosure. For a better understanding of the invention, its operating advantages and specific objects attained by its uses, reference is made to the accompanying drawings and descriptive matter in which preferred embodiments of the invention are illustrated.

#### 20 Detailed description of the figures

Figure 1 is a schematic representation of a preferred embodiment of an apparatus according to the invention for preparing and supplying catalyst to a polymerisation reactor.

Figure 2 is a schematic representation of a single loop polymerisation reactor.

25 Figure 3 is a schematic representation of a double loop polymerisation reactor.

#### Detailed description of the invention

This invention is especially applicable to a process of supplying a catalyst to a polymerisation reactor. The invention is in particular described with reference to the supply of catalyst to a  
30 slurry loop polymerisation reactor wherein ethylene is polymerised. The polymerisation process of ethylene may for instance be carried out in loop reactors. Suitable "ethylene polymerisation" includes but is not limited to homo-polymerisation of ethylene, co-polymerisation of ethylene and a higher 1-olefin co-monomer such as butene, 1-pentene, 1-hexene, 1-octene or 1-decene. Ethylene polymerisation comprises feeding to a reactor the

reactants including the monomer ethylene, a light hydrocarbon diluent, a catalyst and optionally, a co-monomer and hydrogen. In an embodiment of the present invention, said co-monomer is hexene and said diluent is isobutane.

5 In a particularly preferred embodiment, the invention relates to a process of supplying a catalyst to a polymerisation reactor wherein bimodal polyethylene is prepared. "Bimodal PE" refers to PE that is manufactured using two reactors, which are connected to each other in series. However, the present method for improving and optimising catalyst supply to a polymerisation reactor should be understood to be applicable to reactors wherein other types  
10 of polymerisation reactions take place as well.

According to the present invention the term "catalyst" is defined herein as a substance that cause a change in the rate of a polymerisation reaction without itself being consumed in the reaction. Any catalyst allowing ethylene to be polymerised may be used. By way of examples  
15 of such catalysts, mention may be made of catalysts of the Ziegler-Natta type, catalysts based on vanadium or chromium, and metallocene catalysts.

Catalyst slurry can be prepared in different ways. One way consists of preparing catalyst slurry starting from solid catalyst particles, which are suspended in a suitable diluent, e.g. a  
20 hydrocarbon. Generally, such catalyst slurry can be transferred directly to a polymerisation reaction vessel for contact with the monomer reactants.

Catalyst slurry can also be obtained commercially in the form of solid catalyst particles, which are suspended in a mineral oil. Direct injection of such catalyst slurry in the reactor can be  
25 done by connecting the commercial vessel containing the catalyst in oil suspension with the reactor by means of conduits that are provided with suitable pumps. Such pumps typically are suitable for pumping liquids with significant amounts of solids, e.g. solid particles in crude oil. Examples of pumps of this type are commonly known as Moineau pumps or progressive cavity pumps, and are available commercially.

30 Several methods for supplying catalyst to a polymerisation reactor have been described in the prior art. For instance, US 3,846,394 describes a process for the introduction of Ziegler-Natta catalyst slurry in a reactor. The process comprises the preparation of Ziegler-Natta catalyst slurry, the transfer of the slurry via a feed conduit from a storage zone to a metering zone,

and the introduction of the slurry into a reactor. In order to avoid the back flow of monomer and other contents of the reactor into the Ziegler -Natta catalyst conduits the process provides the catalyst feed conduit to be flushed with an inert diluent to the Ziegler -Natta catalyst, said diluent being introduced into said conduit downstream of the metering zone.

5

Catalyst systems for polymerisation and co -polymerisation of olefins known as Ziegler -Natta systems consist on the one hand, as catalyst, of compounds of transition metals belonging to Groups IV to VII of the periodic table of elements, and on the other hand, as co -catalysts, of organometallic compounds of metals of Groups I to III of this Table. The catalysts most frequently used are the halogenated derivatives of titanium and vanadium, preferably associated with compounds of magnesium. Moreover, the co -catalysts most frequently used are organoaluminium or organozinc compounds. A characteristic of all Ziegler -Natta catalysts is that they all yield straight chain polymers.

10

15 The Ziegler-Natta catalyst is preferably of the general formula  $MX_n$  wherein M is a transition metal compound selected from group IV to VII, wherein X is a halogen, and wherein n is the valence of the metal. Preferably, M is a group IV, group V or group VI metal, more preferably titanium, chromium or vanadium and most preferably titanium. Preferably, R is chlorine or bromine, and most preferably, chlorine. Illustrative examples of the transition metal compounds comprise but are not limited to  $TiCl_3$ ,  $TiCl_4$ . In a particularly preferred embodiment of the invention said catalyst is a titanium tetrachloride ( $TiCl_4$ ) catalyst.

20

The term "metallocene catalyst" is used to describe any transition metal complexes consisting of metal atoms "sandwiched" between one or two ligands. In a preferred embodiment, the metallocene catalyst has a general formula  $MX$ , wherein M is a transition metal compound selected from group IV and wherein X is a ligand composed of one or two groups of cyclopentadienyl (Cp), indenyl, fluorenyl or their derivatives. Illustrative examples of metallocene catalysts comprise but are not limited to  $Cp_2ZrCl_2$ ,  $Cp_2TiCl_2$  or  $Cp_2HfCl_2$ .

25

30 The use of metallocene catalysts in the production of polyolefins in general, and of polyethylene in particular, is known in the art. The metallocene catalysts are compounds of Group IV transition metals of the Periodic Table such as titanium, zirconium, hafnium, etc., and have a coordinated structure with a metal compound and ligands composed of one or two groups of cyclopentadienyl, indenyl, fluorenyl or their derivatives. Use of metallocene

catalysts in the polymerisation of olefins has various advantages. Metallocene catalysts have high activities and are capable of preparing polymers with enhanced physical properties in comparison with the polymers prepared using Ziegler -Natta catalysts. Metallocene catalysts are usually employed with a co -catalyst such as an organometallic compound, or a mixture of non-coordinated Lewis acid and alkylaluminium as it is well known in the art. The key to metallocenes is the structure of the complex. The structure and geometry of the metallocene can be varied to adapt to the specific need of the producer depending on the desired polymer. Metallocenes comprise a single metal site, which allows for more control of branching and molecular weight distribution of the polymer. Monomers are inserted between the metal and growing chain of polymer.

Chromium-type catalysts refer to catalysts obtained by deposition of chromium oxide on a support, e.g. a silica or aluminum support. Illustrative examples of chromium catalysts comprise but are not limited to  $\text{CrSiO}_2$  or  $\text{CrAl}_2\text{O}_3$ .

According to a preferred embodiment the catalyst prepared and supplied according to the present invention is a Ziegler-Natta catalyst. In a preferred embodiment, said catalyst is provided as solid particles suspended in a mineral oil.

Generally co-catalysts are used to improve the activity of a Ziegler -Natta or a metallocene catalyst. The term "co-catalyst" as used herein is defined as a catalyst that can be used in conjunction with another catalyst in order to improve the activity and the availability of the other catalyst in a polymerisation reaction. In a preferred embodiment, said co-catalyst is a catalyst suitable for being used in conjunction with a Ziegler -Natta catalyst or a metallocene catalyst. The co-catalyst is used to promote the polymerisation activity of the Ziegler-Natta catalyst or the metallocene catalyst. Broadly, organometallic compounds of periodic groups I to III can be used as co-catalyst according to the present invention. In a particularly preferred embodiment, said co-catalyst is a catalyst suitable for being used in conjunction with a Ziegler-Natta or a metallocene catalyst and is an organoaluminium compound, being optionally halogenated, having general formula  $\text{AlR}_3$  or  $\text{AlR}_2\text{Y}$ , wherein R is an alkyl having 1 - 16 carbon atoms and R may be the same or different and wherein Y is hydrogen or a halogen. Examples of co-catalysts comprise but are not limited to trimethyl aluminum, triethyl aluminum, di-isobutyl aluminum hydride, is tri-isobutyl aluminium, tri-hexyl aluminum, diethyl

aluminum chloride, or diethyl aluminum ethoxide. A particularly preferred co-catalyst for use in the present invention is tri-isobutyl aluminium (TIBAL).

The invention will be described hereunder with reference to a method and apparatus for supplying a Ziegler-Natta catalyst, in particular a titanium tetrachloride ( $\text{TiCl}_4$ ) catalyst to a polymerisation reactor wherein ethylene is polymerised. As co-catalyst for the Ziegler-Natta catalyst, reference is made to a tri-isobutyl aluminium co-catalyst. However, it should be clear that the present apparatus is applicable to other types of catalysts and co-catalysts as well, as indicated above.

As used herein, the term "catalyst slurry" refers to a composition comprising catalyst solid particles that are in suspension. The term "concentrated catalyst slurry" refers to a composition comprising catalyst solid particles that are in suspension whereby the concentration of catalyst is at least higher than 10 % by weight. The term "diluted catalyst slurry" refers to a composition comprising catalyst solid particles that are in suspension, whereby the concentration of catalyst is lower than or equal to 10 % by weight.

The hereunder-described apparatus corresponds to the required equipment for preparation and injection of one catalyst. If two or more (different) catalysts need to be fed to a reactor, two or more apparatuses according to the present invention can be supplied or a catalyst blend can be prepared and supplied using a apparatus according to the present invention.

Referring to FIG.1 a preferred embodiment of an apparatus according to the present invention is illustrated. In general, the apparatus according to the invention comprises a vessel 2 for receiving catalyst in oil suspension, and a buffer vessel 3 for preparing and storing diluted catalyst slurry at a suitable concentration for use in a polymerisation reaction. Concentrated catalyst slurry is transferred from the vessel 2 to the buffer vessel 3 through one or more conduits 4 by means of pumps 5, while diluted catalyst slurry is continuously transferred from the buffer vessel 3 to the reactor through one or more conduits 8 by means of pumps 15. The constructional details of valves, pumps etc. have been omitted in the drawings for clarity, it being within the skill of the art to supply these.

According to the present invention, the Ziegler-Natta catalyst  $\text{TiCl}_4$  is provided as a suspension of solid particles in a mineral oil or hexane or heptane in a commercially available

drum. It can also be supplied in dry form. The catalyst can be transferred to a vessel 2 from this commercial drum. According to an embodiment, the catalyst can be provided from such commercial drums to vessel 2 by means of nitrogen pneumatic transfer or by gravity. The catalyst concentration in vessel 2 can be adjusted by adding mineral oil; alternatively other hydrocarbons can be used.

In general, the pressure in the vessel 2 may comprise approximately between 7 and 16 bar. The slurry in the vessel 2 is referred to as "concentrated" or "heavy" slurry since it contains a proportionally high amount of particulate catalyst solids. Such concentration preferably ranges from 10 to 50 % by weight, and even more preferred from 20 to 40 % by weight.

According to a preferred embodiment, the concentrated Ziegler -Natta catalyst is transferred from vessel 2 to the buffer vessel 3, wherein the catalyst is diluted to a concentration suitable for use in the polymerisation reactor. The buffer vessel 3 therefore is provided with means for supplying a suitable diluent to said buffer vessel 3. The concentrated catalyst supplied to the buffer vessel 3 through conduit 4 is diluted by the diluent supplied through conduit 9 to obtain diluted catalyst slurry in the buffer vessel 3. The buffer vessel 3 can be operated either when full of liquid or not. Preferably, the buffer vessel 3 is operated when full of liquid, since if there is an interphase with nitrogen the catalyst slurry might stick to the walls settle in the vessel.

When using  $\text{TiCl}_4$  as catalyst, hydrocarbons such as hexane or isobutane can be used to dilute the catalyst and to obtain diluted catalyst slurry. However, a major disadvantage of using hexane as diluent to prepare the catalyst is that a portion of hexane generally ends up in the final polymer product, which is undesirable. Isobutane on the other hand is easier to handle, to purify and to re-use in the polymerisation process than hexane. For instance, since in the polymerisation process of ethylene, isobutane is applied as diluent in the reaction, isobutane used as diluent for the catalyst can easily be re-used in the polymerisation process. Therefore, in a preferred embodiment, isobutane is used as diluent for the  $\text{TiCl}_4$  catalyst. Isobutane is generally present in gas form at room temperature and at atmospheric pressure. In order to obtain liquid isobutane for preparing the diluted catalyst slurry, the buffer vessel 3 is preferably operated at pressure levels comprised between 8 and 17 bar, and preferably at pressure levels comprised between 4 and 5 bar. The pressure in the buffer vessel 3 is



preferably lower than the pressure in the reactor, in order to avoid leakage of catalyst from the buffer vessel to the reactor.

Before transferring the Ziegler-Natta catalyst from the vessel 2 to the buffer vessel 3, isobutane is admitted into the vessel 3. The vessel 3 is provided with an inlet system 9 for supply of this diluent. The vessel 2 and the buffer vessel 3 are agitated by means of stirring or mixing means 7 provided in said vessel to maintain the homogeneity of the concentrated and diluted catalyst slurry, respectively. The buffer vessel 3 is preferably large enough to contain sufficient catalyst slurry and large enough such that a day vessel capacity is equivalent to the time to prepare a new batch. This enables to assure the continuous production and availability of the catalyst in the polymerisation reaction. Alternatively, a second vessel 2 can be provided to prepare a new batch.

The slurry in the buffer vessel 3 is referred to as "diluted" slurry since it contains a proportionally low amount of particulate catalyst solids. The diluted slurry has a concentration comprised between 0.1 and 10 % by weight, and preferably comprised between 0.1 and 5 % by weight, and even more preferred between 0.5 and 4 % by weight. Preparing diluted catalyst slurry having these concentrations advantageously enables the further use of diaphragm pumps 15 for injecting the diluted catalyst slurry in the reactor 1, as described in more detail below. In case other concentrations of catalyst slurry would be applied, it is clear that other types of pumps can be applied.

The transfer of the Ziegler-Natta catalyst from the vessel 2 to the buffer vessel 3 is preferably done through one or more conduits 4. For transfer of Ziegler-Natta catalyst from the vessel 2 to the buffer vessel 3 a pump 5 is provided on each conduit 4. In a preferred embodiment, said pump 5 comprises a pump which is suitable for pumping liquids with significant amounts of solids, e.g. solid particles in crude oil, which would otherwise easily damage more common types of reciprocating oil well pumping systems. Examples of pumps of this type are commonly known as Moineau pumps or progressive cavity pumps, and are available commercially. Such progressive cavity pumps operate on the Moineau principle, which is based on the geometrical fit between the rotating element (rotor), and the stationary element (stator). The interference fit between the rotor and the stator creates a series of sealed chambers called cavities. Pumping action is achieved by the rotor turning eccentrically within the stator. Fluid enters the cavity formed at the inlet and progresses within that cavity to the

outlet. The result is a positive displacement, non-pulsating flow that is directly proportional to the pump's speed. This allows the progressive cavity pump to deliver material at a wide range of flow rates from small shots to continuous flow.

- 5 As represented in FIG. 1, the conduits 4 preferably leave the vessel 2 in upward direction under an angle preferably superior to  $10^\circ$ , and more preferably superior to  $30^\circ$ . In addition, the conduits 4 provided downwards the pumping means 5 conducts the catalyst slurry preferably downwardly, under an angle preferably superior to  $10^\circ$ . Such configuration improves the action of the pump 5 and also enables to avoid plugging in the pump 5 since  
 10 under this configuration the concentrated catalyst slurry tends to settle away from the pumps 5 in case the pumps 5 are interrupted or stopped.

- The conduits 4 are further preferably provided with a pulsation dampener, safety valves and isobutane flushing means 10, either at the inlet, at the outlet or at both sides of the slurry  
 15 pumps 5, as illustrated on FIG. 1. Isobutane flushing means 10 enable to flush isobutane diluent through the conduit 4 and to keep the conduits 4 and the pumps 5 unplugged. On conduits 10 for injecting isobutane flow measuring means can be provided. When different conduits 4 are provided for connecting the vessel 2 to the buffer vessel 3, generally, one conduit having one active pump 5 will be operative, while the other conduits 4 and pumps 5  
 20 will not be operative but will be kept in stand by mode.

- The concentrated slurry is preferably injected in ratio control of isobutane diluent to catalyst in the buffer vessel to have a constant concentration of slurry in the buffer vessel. In addition, the conduits 4 can be further provided with flow measuring means for easily measuring the  
 25 flow rate of the concentrated catalyst slurry in the conduits 4. The ratio of catalyst to diluent is adequately controlled and adjusted by controlling the speed of the pump 5 and by measuring the density of the isobutane diluent.

- Catalyst wastes can be sent through conduit 17, which is provided with a valve 20, to one or  
 30 more dump vessels 18, which are preferably provided with stirring means and contain mineral oil for neutralization and elimination of the wastes. Preferably said dump vessel 18 is larger than the buffer vessel 3. In case of the preparation of unsuitable catalyst, these can be emptied from the vessels 3 to these dump vessels 18. The dump vessel 18 is preferably a heated vessel, having a steam jacket, where the diluent, i.e. isobutane, is evaporated. The

steam jacket is preferred, for desorbing isobutane. The evaporated diluent is sent to distillation or to the flare. In order to avoid the transfer of catalyst fragments when transferring the evaporated diluent, guard filters are provided with the dump vessels 18. The dump vessels 18 are also provided with pressure controlling means for controlling the pressure in said vessels. The catalyst waste remaining after evaporation of the diluent is removed from the vessels 18, preferably by means of a draining system, provided at the bottom of the vessel 18, and the removed waste is discharged into drums and further destroyed.

The diluted Ziegler-Natta catalyst slurry is subsequently transferred from the buffer vessel 3 to the reactor 1 through one or more conduits 8. The conduits 8 preferably have an inner diameter comprised between 0.3 and 2 cm, and preferably between 0.6 and 1 cm. Each conduit 8 is provided with a pump 15, which controls the transfer and injection of the diluted Ziegler-Natta catalyst slurry into the reactors 1. In a particularly preferred embodiment, said pumps are diaphragm pumps.

As represented in FIG. 1, the conduits 8 preferably leave the buffer vessel 3 upwardly under an angle preferably superior to  $10^\circ$ , and more preferably superior to  $30^\circ$  preferably superior to  $10^\circ$ . In addition, the conduit 8 provided downwards the pump 15 conducts the diluted catalyst slurry downwardly, under an angle preferably superior to  $10^\circ$ . Such configuration improves the action of the pump 15 and also enables to avoid plugging in the pump 15 since under this configuration the diluted catalyst slurry tends to settle away from the pumps 15 in case the pumps 15 are interrupted or stopped.

The conduits 8 are further provided with a pulsation dampener, safety valves and isobutane flushing means 19, either at the inlet, at the outlet or at both sides of the diaphragm pumps 15. Isobutane flushing means 19 enable to flush isobutane through the conduit 8 and to keep the conduits 8 and the pump 15 unplugged. Preferably, there is continuous flushing of the conduit 8 downstream the membrane pump 15 to the reactor 1 by means of isobutane flushing means. The conduit 8 upstream the pump 15 may be flushed discontinuously, by means of isobutane flushing means. When different conduits 8 are provided for connecting the buffer vessel 3 to the reactor 1, generally, one conduit having one active pumps 15 will be operative, while the other conduits 8 and pump 15 will not be operative but will be kept in stand by mode. In this latter case, the conduit 8 downstream the pump 15 will preferably be

flushed with a suitable stream of diluent. The conduit 8 upstream the pump 15 may be discontinuously flushed.

5 In order to reduce the risk of leakage, the catalyst should be stored at a lower pressure than the reactor that is generally comprised around 43 bar, e.g. stored in the buffer vessel 3 at approximately 6-16 bar. The pressure in the conduits 8 downstream of the pumps 15 is preferably comprised between 45 and 65 bar. This elevated pressure, in comparison with the pressure values provided in the vessel 2 and the buffer vessel 3, is required in order to bring the diluted catalyst under sufficient pressure into the reactor.

10 It is important to control correctly the catalyst flow to the reactor and to pump catalyst slurry into the reactor at a controlled and limited flow. An unexpected flow to the reactor could lead to a runaway reaction. A fluctuating flow to the reactor could lead to reduced efficiency and fluctuations in product quality. Therefore, in a particularly preferred embodiment, the injection  
15 pump 15 flow rates are controlled by the reactors' 1 activity. The pumps are in particular controllable in function of the concentration of a reactant in said reactor. Preferably said reactant is the concentration of monomer, i.e. ethylene, in the reactor. However, it should be clear that the membrane pumps are controllable in function of the concentration of other reactants, such as e.g. the co-monomer or hydrogen concentrations in the reactor as well. By  
20 the use of membrane pumps 15 the invention provides for a good control of the diluted catalyst flow. In particular, the catalyst flow rate to the reactors is controlled by adjusting the stroke and/or frequency of the diaphragm pumps. Furthermore, the pump flow rates are controlled by the ethylene concentration in the reactor. In case the ethylene concentration is high in the reactor, more catalyst will be added to the reactor and vice versa. In this way, the  
25 variations in ethylene polymerisation rate are taken into account and actual production rate and product properties do not fluctuate significantly.

Another problem relating to the field of catalyst supply to a reactor consists of supplying a co-catalyst during a polymerisation reaction. A number of techniques for the introduction of the  
30 co-catalyst have already been proposed, for example by introducing the co-catalyst directly into the polymerisation reactor. However, such methods do not allow bringing co-catalyst into contact with the catalyst before entering the reactor, although such pre-contact may be particularly desirable in order to provide effective catalyst-co-catalyst mixtures. Another technique consists of contacting the catalyst and co-catalyst before their introduction into the

polymerisation medium. In this latter case, however, it is difficult to control the pre -contact time of the catalyst with the co -catalyst.

5 In a further embodiment, the present apparatus is further provided with a co-catalyst distribution system, for bringing a suitable amount of co -catalyst into contact with the catalyst slurry for a suitable period of time before supplying said catalyst slurry to said reactor. When using a Ziegler-Natta catalyst, tri isobutyl aluminium (TIBAL) is preferably used as co -catalyst.

10 The co-catalyst distribution system 12 comprises at least one co -catalyst storage vessel wherein co-catalyst is prepared and stored and a conduit 11 connected thereto for transferring said co -catalyst, as illustrated on FIG. 1. Co-catalysts are generally provided in commercial drums. In a storage vessel of the co -catalyst distribution system 13, the TIBAL co-catalyst is generally provided in a solution of hexane or heptane, but can be provided in pure form too. The TIBAL co -catalyst is transferred from the storage vessel through a co -  
15 catalyst injection conduit 11, in the conduit 8, which connects the buffer vessel 3 with the reactor 1. Conduit 11 intersects conduit 8, downstream the diaphragm pumps 15 and upstream the reactor 1.

20 Co-catalyst wastes can be sent to a dump vessel, which is preferably provided with stirring means and contains mineral oil for neutralization and elimination. The dump is provided with a heated vessel, e.g. steam jacket, where the isobutane is evaporated and sent to distillation or to the flare.

25 The contact time between the Ziegler -Natta catalyst and the TIBAL co -catalyst and the ratio between the Ziegler-Natta catalyst and the TIBAL co -catalyst have an important influence on the granulometry but also on the activity of the final polymerisation product. Using a TIBAL co-catalyst, bigger polyethylene particles can be obtained through activity. Also, pre -contact of the TIBAL co-catalyst with the Ziegler-Natta catalyst improves the bulk density and the settling efficiency of the polyethylene prepared in the polymerisation reactor. According to the  
30 invention a suitable amount of TIBAL co -catalyst is injected in the conduits 8, downstream the diaphragm pumps 15, before entering the reactors 1.

In case the TIBAL co -catalyst is injected in the conduit 8, the injection point is at a distance from the reactor allowing a certain pre -contact time with the catalyst before being supplied to

the reactor. In order to have a sufficient pre-contact time, preferably between 5 seconds and 1 minute, between the Ziegler-Natta catalyst slurry and the TIBAL co-catalyst, each conduit 8 is provided with a contact vessel 13, preferably downstream the injection point of the co-catalyst distribution system, for enhancing the contact time of said co-catalyst with said catalyst slurry in the conduits 8. These contact vessels can be agitated or not. In another preferred embodiment, the conduits 8 have an inner diameter comprised between 0.3 and 2 cm, and preferably comprised between 0.6 and 1 cm while the diameter of the contact vessels 13 is preferably comprised between 1 and 15 cm and preferably between 6 and 9 cm.

In addition, at least one flow measuring means 16 is further provided on conduits 8 for easily measuring the catalyst slurry flow rate in the conduits 8. These flow measuring means 16 preferably are Coriolis flow measuring means that are preferably provided downstream from said diaphragm pumps 15. The flow measuring means 16 can however also be provided between the buffer vessel 3 and the membrane pumps 5. Preferably, said means 16 are provided upstream of the co-catalyst injection conduit 11. The diluted slurry is preferably injected in ratio control of isobutane diluent to catalyst. The ratio of catalyst to diluent is adequately controlled and adjusted by controlling the speed of the pump 15 and by measuring the density of the isobutane diluent. The Coriolis meters 16 can measure the flow and the density of the catalyst slurry at the exit of the buffer vessel 3 and indirectly determine the suspended solids concentration. A correlation exists for estimating the concentration of suspended solids based on the slurry density, the carrier fluid density and the solid particle density.

In another embodiment, the catalyst slurry is injected under controlled flow rate into the reactor. The conduits 8 for transferring catalyst slurry into to the reactor are equipped by one or more valves, preferably piston valves 14. The piston valves 14 are capable of sealing the orifice by which the conduit 8 is connected to the reactor 1. When using different conduits 8 for transferring catalyst slurry to one reactor, only in one conduit 8 the pumps actively pump catalyst slurry to the reactor, while in other conduits 8 the pumps are not active and the conduits are preferably flushed by isobutane.

The apparatus according to the invention can be applied for feeding a single polymerisation reactor. In a preferred embodiment the apparatus according to the invention is applied for feeding a polymerisation reactor consisting of two liquid full loop reactors, comprising a first

and a second reactor connected in series by one or more settling legs of the first reactor connected for discharge of slurry from the first reactor to said second reactor. Such in series connected reactors are particularly suitable for the preparation of bimodal polyethylene. The present apparatus, comprising the vessel 2, buffer vessel 3, the conduits 4 and 8, each provided with their respective pumps, the co-catalyst distribution system 12, and optionally one or more dump vessels 18, can be applied for both reactors. The number of conduits 8 can be divided between the first and the second reactor. It is also possible to use two or more apparatuses according to the invention, e.g. when two or more different catalysts are used.

10 It is clear from the present description that numbers and dimensions of the different parts of the apparatus according to the present invention relate to the size of the polymerisation reactors and can be changed in function of the reactor sizes.

15 In another preferred embodiment, by operation in accordance with the present invention, all lines, vessels, pumps, valves, etc.. can be kept free of clogging by means of flushing or purging with nitrogen or diluent, i.e. isobutane. It is to be understood that where necessary flushing and purging means and lines are available on the apparatus according to the invention in order to avoid plugging or blocking.

20 It is to be understood from the present invention that all indicated pressure values are preferred pressure values, which in general can deviate from the indicated pressure values with approximately  $\pm 1$  bar. It will be evident from the present description that all indicated pressure values in the vessels, conduits, etc.. are values that are lower than the pressure value in the polymerisation reactor.

25 In another preferred embodiment, it is to be understood that all lines or conduits applied in accordance with the present invention may be provided, where necessary with flow measuring means.

30 In a preferred embodiment, the device according to the invention can be used in a single loop reactor as depicted on FIG. 2 or in a double loop reactor, as depicted on FIG. 3.

**FIG. 2** represents a single loop reactor 100, consisting of a plurality of interconnected pipes 104. The vertical sections of the pipe segments 104 are preferably provided with heat jackets 105. Polymerisation heat can be extracted by means of cooling water circulating in these jackets of the reactor. Reactants are introduced into the reactor 100 by line 107. Catalyst, optionally in conjunction with a co-catalyst or activation agent, is injected in the reactor 100 by means of the conduct 106. The polymerisation slurry is directionally circulated throughout the loop reactor 100 as illustrated by the arrows 108 by one or more pumps, such as axial flow pump 101. The pump may be powered by an electric motor 102. As used here in the term "pump" includes any device from compressing driving, raising the pressure of a fluid, by means for example of a piston or set of rotating impellers 103. The reactor 100 is further provided with one or more settling legs 109 connected to the pipes 104 of the reactor 100. The settling legs 109 are preferably provided with an isolation valve 110. These valves 110 are open under normal conditions and can be closed for example to isolate a settling leg from operation. Further the settling legs can be provided with product take off or discharge valves 111. The discharge valve 111 may be any type of valve, which can permit continuous or periodical discharge of polymer slurry, when it is fully open. Polymer slurry settled in the settling legs 109 may be removed by means of one or more product recovery lines 113, e.g. to a product recovery zone.

**FIG. 3** represents two single loop reactors 100, 116, which are interconnected in series. Both reactors 100, 116 consist of a plurality of interconnected pipes 104. The vertical sections of the pipe segments 104 are preferably provided with heat jackets 105. Reactants are introduced into the reactors 100 by line 107. Catalyst, optionally in conjunction with a co-catalyst or activation agent, is injected in the reactor 100 or 116 by means of the conduct 106. The polymerisation slurry is directionally circulated throughout the loop reactors 100, 116 as illustrated by the arrows 108 by one or more pumps, such as axial flow pump 101. The pumps may be powered by an electric motor 102. The pumps may be provided with set of rotating impellers 103. The reactors 100, 116 are further provided with one or more settling legs 109 connected to the pipes 104 of the reactors 100, 116. The settling legs 109 are preferably provided with an isolation valve 110. Further the settling legs can be provided with product take off or discharge valves 111. Downstream the valve 111 at the exit of the settling leg 109 of reactor 100, a three-way valve 114 is provided which allows to transfer polymer slurry settled in the settling legs 109 to the other reactor 116, by means of the transfer line 112. The transfer line 112 connects the three-way valve 114, provided at the exit of the settling leg 109



of one reactor 100, with the entry in the other reactor 116, where preferably a piston valve 115 is provided. Polymer slurry settled in the settling legs 109 of reactor 116 can be removed by means of one or more product recovery lines 113, e.g. to a product recovery zone.

5 In another embodiment, the present invention relates to a method for optimising catalyst supply to a polymerisation reactor 1 comprising the steps of a) transferring concentrated catalyst slurry from a vessel 2 to a buffer vessel 3, said concentrated catalyst slurry comprising catalyst solid particles suspended in a mineral oil, b) diluting said catalyst slurry in  
10 said buffer vessel 3 by supplying a suitable diluent in said buffer vessel 3 whereby diluted catalyst slurry is obtained having a suitable concentration for use in a polymerisation reaction, and c) transferring said diluted catalyst slurry from said buffer vessel 3 to said reactor 1 at a suitable flow rate.

15 Preferably, said method is a method for optimising catalyst supply to a polymerisation reactor 1 wherein polyethylene, and preferably bimodal polyethylene, is prepared.

According to the present invention, the Ziegler-Natta catalyst  $\text{TiCl}_4$  is provided in a commercial container 2 and transferred to vessel 3 as a suspension of solid particles in a mineral oil. The method comprises the step of transferring concentrated catalyst slurry from a  
20 vessel 2 to a buffer vessel 3 through conduits 4 provided with pumps 5, preferably progressive cavity pumps.

In the buffer vessel 3 the catalyst slurry is diluted to a suitable concentration, preferably between 0.1 and 10 % by weight, and more preferred having a concentration comprised  
25 between 0.1 and 5 % by weight, and even more preferred between 0.5 and 4 % by weight. by adding diluent, isobutane, to the vessel 3. Preparing diluted catalyst slurry having these concentrations advantageously enables the further use of diaphragm pumps 15 for injecting the diluted catalyst slurry in the reactor 1. The amount of isobutane diluent can be controlled using the catalyst concentration determined from the density measurement by Coriolis meter  
30 16.

In another embodiment, the method according to the invention comprises controlling the flow rate of the catalyst slurry to the reactor 1 by determining the concentration of a reactant in said reactor 1. Preferably said reactant is the concentration of monomer, i.e. ethylene, in the

reactor. However, it should be clear that also determination of other reactants, such as e.g. the co-monomer or the diluent concentrations in the reactor, is comprised within the scope of the present invention. Practically, this mechanism is obtained by providing each conduit for transferring and supplying the catalyst slurry from the buffer vessel to the reactor with a  
 5 membrane pump that is capable of being adjusted and regulating the catalyst flow rate in function of the concentration of a reactant in said reactor.

In certain cases it might be required or advantageous to bring the catalyst into contact with a co-catalyst, as indicated above. Therefore, the present invention further provides a method  
 10 comprising the step of bringing a co-catalyst into contact with said catalyst slurry before supplying said catalyst slurry to said reactor. The present method provides a better contact and formation of a co-catalyst-catalyst mixture than in the case when co-catalyst is directly supplied to a reactor. Supply of a suitable co-catalyst-catalyst mixture to the reactor provides a more controlled and more uniform level of polymerisation reactivity in the reactor. Also, pre-  
 15 contact between catalyst and co-catalyst positively influences the granulometry of the final polymerisation product and improves the bulk density and the settling efficiency of the polymerisation product prepared in the polymerisation reactor. Such method also enables to more precisely control the ratio of catalyst-co-catalyst injection.

20 In a preferred embodiment, the method comprises bringing a co-catalyst, preferably a co-catalyst as defined above, into contact with said diluted catalyst slurry present in conduits 8. The co-catalyst distribution system 12 preferably comprises at least one storage vessel and a conduit 11 intersecting the conduit 8. In another preferred embodiment, the method further comprises enhancing the contact time and the pre-contact of said co-catalyst with said  
 25 catalyst slurry in conduits 8, by locally enhancing the volume of said conduits 8. Pre-contact between catalyst and co-catalyst positively influences the granulometry of the final polymerisation product and improves the bulk density and the settling efficiency of the polymerisation product prepared in the polymerisation reactor. During the activation process, if too much co-catalyst contacts the catalyst particle, the catalytic activity is not only reduced,  
 30 but actual harm may result. The present method also advantageously enables to more precisely control the ratio of catalyst/co-catalyst injection.

In another preferred embodiment, the present invention provides a method for continuously supplying catalyst slurry from the buffer vessel 3 to the reactor 1 through conduits 8 at a

suitable flow rate. The present invention provides a method that enables to continuously supply catalyst to a reactor, without interruption of the catalyst flow. By this mechanism continuous supply of catalyst slurry without relevant fluctuations to a polymerisation reactor is ensured, which will increase the efficiency of the polymerisation reaction in the reactor.

5

In yet another preferred embodiment, the invention relates to a method wherein catalyst flow rate to the reactor is accurately measured, through liquid flow rate measurement, using flow measuring means, such as preferably Coriolis flow measuring means.

- 10 The present invention also relates to the use of an apparatus according to the present invention for preparing and optimising the supply of a Ziegler-Natta catalyst to a polymerisation reactor wherein polyethylene, and preferably bimodal polyethylene, is prepared.
- 15 While the invention has been described in terms of the presently preferred embodiment, reasonable variations and modifications are possible by those skilled in the art and such variations are within the scope of the described invention and the appended claims.

**Claims**

1. Apparatus for preparing and supplying catalyst to an ethylene slurry loop polymerisation reactor (1) comprising

5 a vessel (2) suitable for containing concentrated catalyst slurry comprising catalyst solid particles suspended in a mineral oil,

a buffer vessel (3) for diluting said catalyst slurry at a suitable concentration for use in a polymerisation reaction, being in connection with said vessel (2) by means of one or more conduits (4) for transferring the concentrated catalyst slurry from said vessel (2) to the buffer vessel (3) and being provided with one or more conduits (8) for transferring the diluted catalyst slurry from said buffer vessel (3) to said reactor (1),

10 a pump (5) provided on each of said conduits (4) for transferring catalyst slurry from said vessel (2) to said buffer vessel (3) and

15 a pump (15) provided on each of said conduits (8) for transferring diluted catalyst slurry from said buffer vessel (3) to said reactor (1).

2. Apparatus according to claim 1, wherein said pump (5) provided on each conduit (4) for transferring the concentrated catalyst slurry from said vessel (2) to the buffer vessel (3) comprises a progressive cavity pump.

20 3. Apparatus according to claim 1, wherein said pump (15) provided on each conduit (8) for transferring the diluted catalyst slurry from said buffer vessel (3) to said reactor (1), comprises a membrane pump, which is controllable in function of the concentration of a reactant in said reactor (1).

25 4. Apparatus according to any of claims 1-3, further comprising a co-catalyst distribution system (12), for bringing a suitable amount of co-catalyst into contact with the catalyst slurry for a suitable period of time before supplying said catalyst slurry to said reactor, said system comprising at least one co-catalyst storage vessel and a conduit (11) connected thereto for  
30 transferring said co-catalyst.

5. Apparatus according to claim 1 or 4, wherein said conduit (8) is provided with a contact vessel (13) for enhancing the contact time of said co-catalyst with said catalyst slurry in said conduit (8).

6. Apparatus according to any of claims 1-5, further comprising flow measuring means (6) provided on conduit (4) and flow measuring means (16) provided on conduit (8) for measuring the catalyst flow rate.

7. Apparatus according to any of claims 1-6, wherein said polymerisation reactor (1) is suitable for preparing polyethylene, and preferably for preparing bimodal polyethylene.

8 Device according to any of claims 1-7, wherein said catalyst is a Ziegler-Natta catalyst having general formula  $MX_n$ , wherein M is a transition metal compound selected from group IV to VII, wherein X is a halogen, and wherein n is the valence of the metal.

9. Apparatus according to any of claims 4-5, wherein said co-catalyst is an organo-aluminium compound, being optionally halogenated, having general formula  $AlR_3$  or  $AlR_2Y$ , wherein R is an alkyl having 1-16 carbon atoms and R may be the same or different and wherein Y is hydrogen or a halogen.

10. Method for optimising catalyst supply to an ethylene slurry loop polymerisation reactor (1) comprising the steps of

- a) transferring concentrated catalyst slurry from a vessel (2) to a buffer vessel (3), said concentrated catalyst slurry comprising catalyst solid particles suspended in a mineral oil,
- b) diluting said catalyst slurry in said buffer vessel (3) by supplying a suitable diluent in said buffer vessel (3) whereby diluted catalyst slurry is obtained having a suitable concentration for use in a polymerisation reaction, and
- c) transferring said diluted catalyst slurry from said buffer vessel (3) to said reactor (1) at a suitable flow rate.

11. Method according to claim 10 for optimising catalyst supply to a polymerisation reactor (1) wherein polyethylene, and preferably bimodal polyethylene, is prepared.

12. Method according to claim 10 or 11, wherein said catalyst is a Ziegler-Natta catalyst having general formula  $MX_n$ , wherein M is a transition metal compound selected from group IV to VII, wherein X is a halogen, and wherein n is the valence of the metal.

13. Method according to any of claims 10 -12, comprising controlling the catalyst flow rate of said catalyst slurry to said reactor by determining the concentration of a reactant, preferably ethylene, in said reactor (1).

5

14. Method according to claim 10, further comprising bringing a suitable amount of co -catalyst into contact with the catalyst slurry for a suitable period of time before supplying said catalyst slurry to said reactor.

10

15. Method according to claim 14, wherein said co-catalyst is an organoaluminium compound, being optionally halogenated, having general formula  $AlR_3$  or  $AlR_2Y$ , wherein R is an alkyl having 1-16 carbon atoms and R may be the same or different and wherein Y is hydrogen or a halogen.

15

16. Method according to claim 14 or 15, comprising bringing a co -catalyst into contact with said catalyst slurry present in the conduit (8).

17. Method according to claim 16, comprising enhancing the contact time of said co -catalyst with said catalyst slurry in the conduit (8), by locally enhancing the volume of said conduits (8).

20

18. Method according to any of claims 10-17, comprising continuously supplying said catalyst slurry from said buffer vessel (3) to said reactor (1) through conduits (8) at a suitable flow rate.

25

19. Use of an apparatus according to any of claims 1 -9 for preparing and optimising the supply of a Ziegler-Natta catalyst to a polymerisation reactor (1) wherein polyethylene, and preferably bimodal polyethylene, is prepared .



**Abstract****Method and apparatus for preparing and supplying catalyst slurry to a polymerisation reactor**

5

The invention relates to an apparatus for preparing and supplying catalyst to an ethylene slurry loop polymerisation reactor (1) comprising a vessel (2) suitable for containing concentrated catalyst slurry comprising catalyst solid particles suspended in a mineral oil, a buffer vessel (3) for diluting said catalyst slurry at a suitable concentration for use in a polymerisation reaction, being in connection with said vessel (2) by means of one or more conduits (4) for transferring the concentrated catalyst slurry from said vessel (2) to the buffer vessel (3) and being provided with one or more conduits (8) for transferring the diluted catalyst slurry from said buffer vessel (3) to said reactor (1), a pump (5) provided on each of said conduits (4) for transferring catalyst slurry from said vessel (2) to said buffer vessel (3) and a pump (15) provided on each of said conduits (8) for transferring diluted catalyst slurry from said buffer vessel (3) to said reactor (1). The invention further relates to a method for optimising catalyst supply to a polymerisation reactor.

20





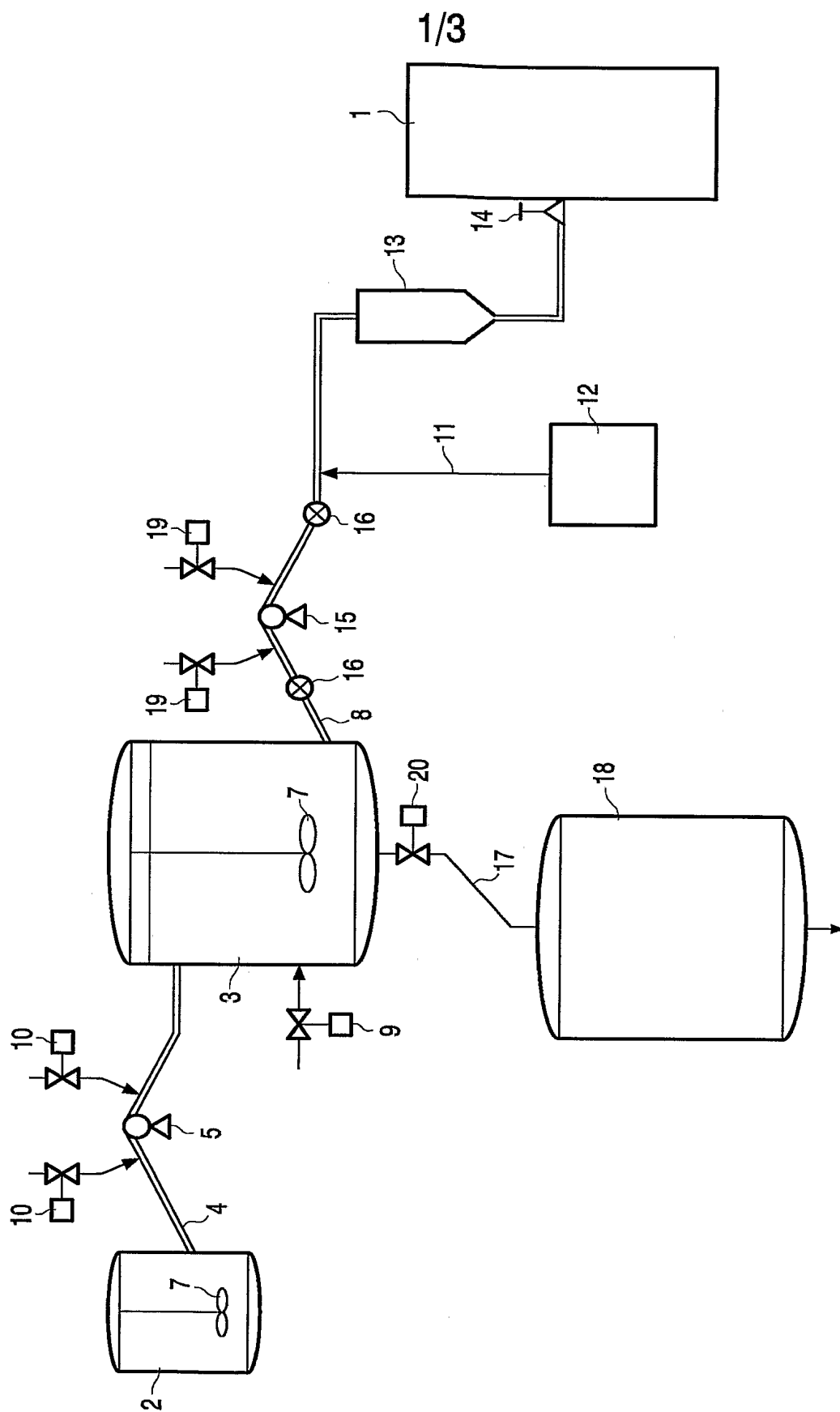


FIG. 1

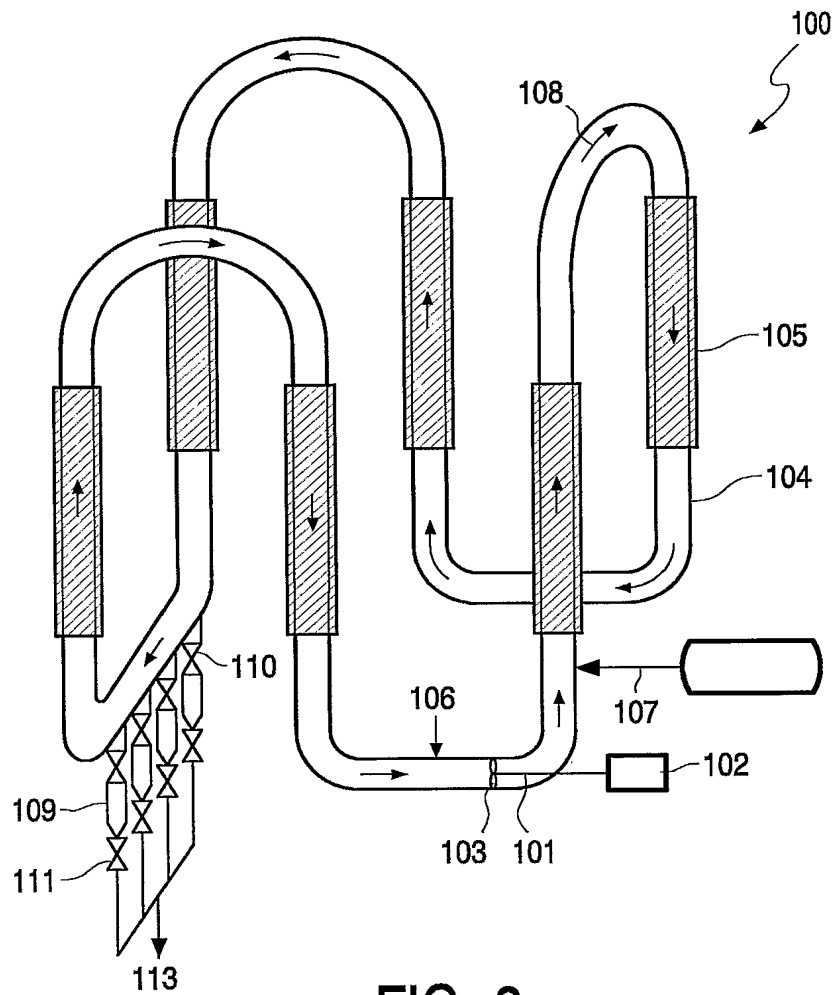


FIG. 2

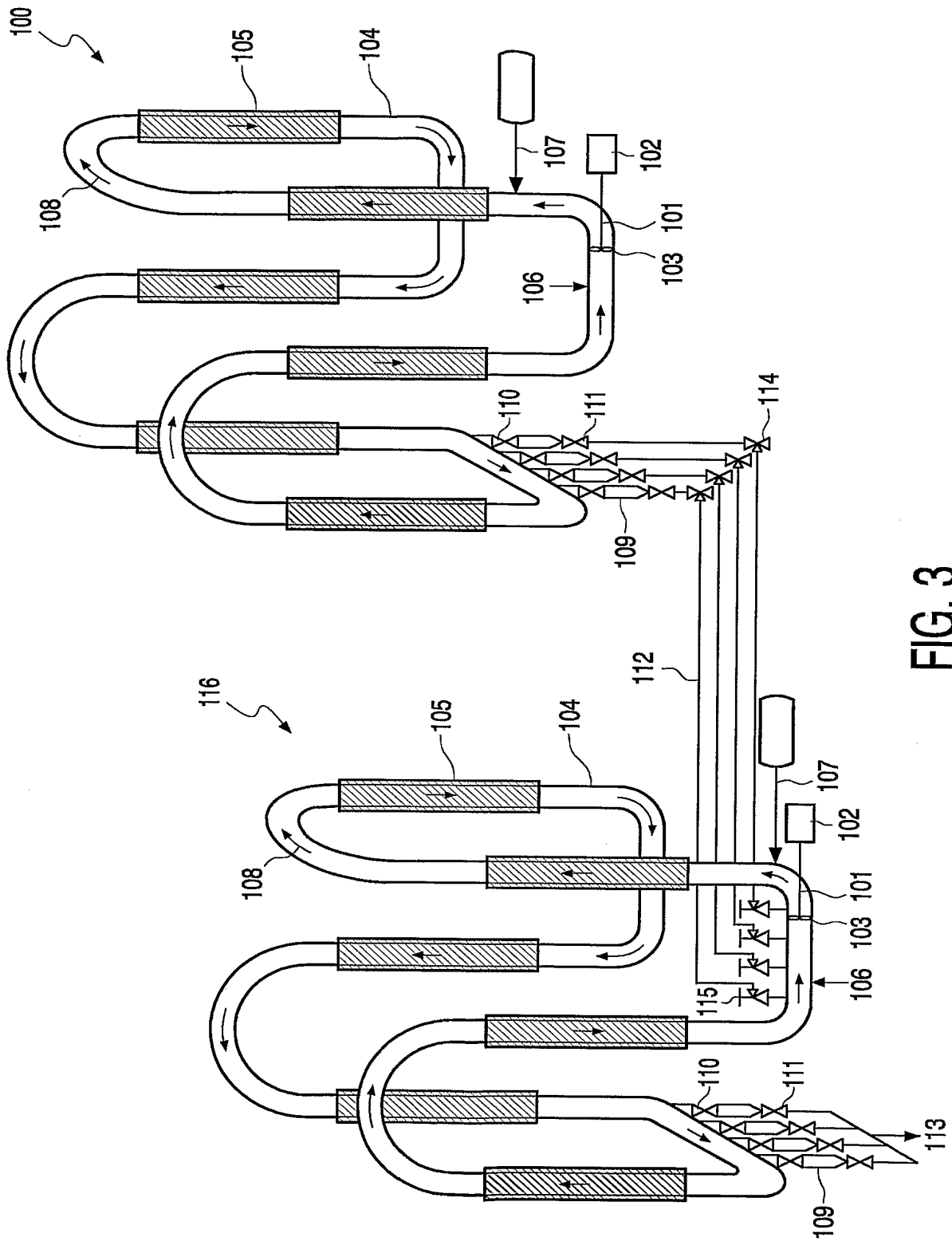


FIG. 3

